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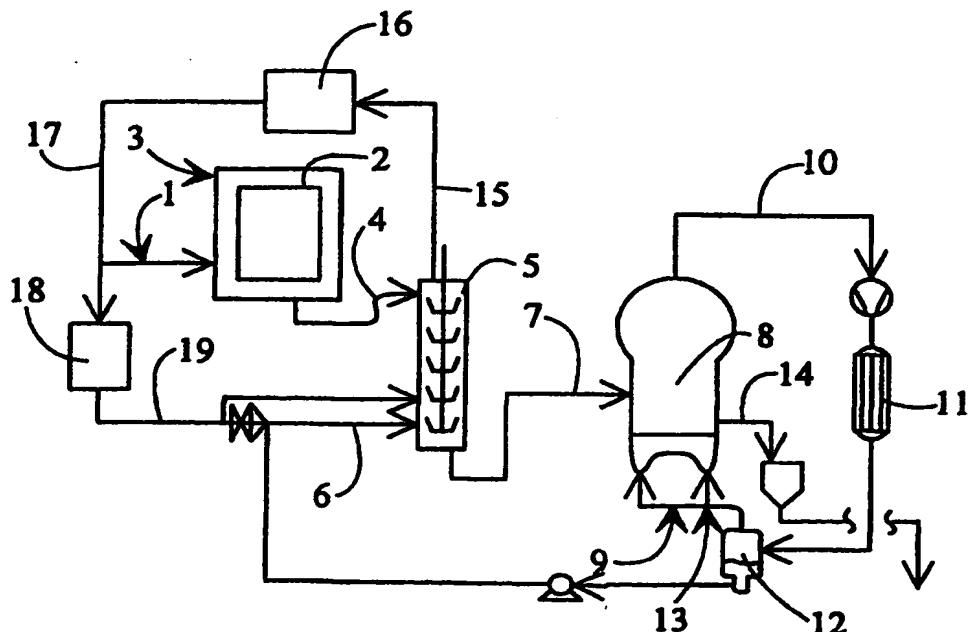
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**(54) Title: METHOD FOR TRANSFERRING POLYMER INTO A GAS PHASE REACTOR**



**(57) Abstract**

The invention concerns a method for transferring polymer from a slurry reactor into a gas phase reactor. Polymer suspended in the liquid medium in the slurry reactor is separated, mixed with a second liquid medium, mixed with a third liquid medium, and transferred with said third medium into the gas phase reactor.

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## METHOD FOR TRANSFERRING POLYMER INTO A GAS PHASE REACTOR

### Field of the invention

5 The present invention relates to polymer production and concerns a method for transferring polymer from a liquid slurry reactor to a gas phase reactor in a multi-stage polymerization process. The invention can be used especially in polyolefin production.

### Background of the invention

10 Polymers are often prepared in a multistage process in order to provide desired properties. Especially molecular weight distribution can thus be controlled. The process may comprise, for example, a liquid phase polymerization and thereafter a gas phase polymerization. Such two-stage processes have been used especially for preparing polyolefins, such as polyethylene.

15 US-A-4368291 discloses a process for preparing polyethylene, in which ethylene is first polymerized in a liquid reactor and thereafter in a gas phase reactor. Liquid hydrocarbon is used as a medium in the first stage, and the liquid medium and polymer together are transferred to the second stage. Dispersion medium from the gas phase reactor can be removed and cooled, possibly condensed, and recycled into the reactor. This provides an effective method to control the temperature in the reactor. One disadvantage of this process is that the medium of the first stage may disturb the polymerization reactions in the second stage. For example, hydrogen from the first stage may be harmful in the second stage.

20 US-A-4692501 discloses a two-stage polymerization process where the first stage is conducted as a slurry polymerization in a continuous stirred tank reactor and the second stage is conducted in a gas phase reactor. Between the reactors there is an liquid exchanging stage. The slurry from the first polymerization stage is introduced into the top of the liquid medium exchanging zone and fresh liquid is introduced into the bottom of liquid exchanging stage. The slurry containing the fresh liquid and the polymer is then collected from the bottom of the zone and introduced into a gas phase reactor. The slurry medium from the first polymerization stage is collected from the top of the zone. The amount of fresh liquid introduced into the bottom is such that the volume of liquid discharged with the polymer from the liquid

exchanging zone is 1 to 8 times, preferably 1.05 to 1.5 times the volume of the fresh liquid.

WO-A-92/12182 (based on FI-A-906428) discloses a two-stage process for preparing polyethylene, in which the first stage is a liquid phase polymerization in a 5 loop reactor. The polyethylene powder prepared in the first polymerization stage is separated from the liquid phase containing the diluent, monomer(s) and hydrogen in a flash separation stage. Thus the liquid medium of the first stage does not disturb the gas phase reaction. However, the transfer of polymer powder may be difficult in some cases.

10 To ensure a smooth operation of a gas phase reactor, the polymer powder should contain only a small fraction of polymer particles having a small diameter. These small particles are often referred to as fines. If the fines are present in a high amount, so that more than about 15% of the particles have a diameter smaller than 100 microns, problems in the operation of the gas phase reactor and/or product 15 transfer are likely. It has been found that the fines are present already after the first polymerization stage. Different methods to reduce the level of fines have been presented, like the use of a prepolymerization reactor, as disclosed in WO-A-96/18662 (based on FI-A-945926).

#### General description of the invention

20 Now a method according to claim 1, a process according to claim 2, and a plant according to claim 11 have been invented. Some preferred embodiments of the invention are defined in the dependent claims.

According to the invention, the polymer is prepared in the first stage in the presence 25 of a first liquid medium comprising diluent with monomer(s) and possibly hydrogen dissolved therein. The polymer is separated from the first liquid medium and mixed in a second liquid medium. This is suitably done in a washing apparatus. After the separation, the polymer powder is then transferred with a third liquid medium into the gas phase reactor. Thus no dry polymer powder needs to be transferred.

30 The second liquid medium may be the same as or different from the diluent used in the first polymerization stage. Preferably, the second and third liquid mediums have such boiling point and vapor pressure that they evaporate in the conditions employed in the second polymerization stage in the gas phase reactor. The third liquid medium is most preferably obtained by condensing a part of the recycle gas of the gas phase reactor.

It has been found that in a process where the first polymerization stage is conducted in a slurry reactor and the second polymerization stage in a gas phase reactor and the hydrocarbons are separated from the polymer after the slurry reactor by flashing, the fines are mainly produced at the flash stage. The particles break when the 5 pressure is reduced and the hydrocarbons are released from the particles. In accordance with the invention, the formation of polymer fines is minimized, when there is no flash stage.

The invention can be used especially in the polymerization or copolymerization of 10 olefins, such as ethylene or propylene or their mixture, optionally together with higher alpha-olefins.

The first stage is preferably carried out in a loop reactor, and the second stage in a fluidized bed reactor.

#### **Brief description of the drawings**

The enclosed drawings form a part of the written description.

15 Figure 1 is a schematic representation of a two-stage polymerization process.

#### **Detailed description of the invention**

The first polymerization stage is a slurry polymerization, and it is preferably carried out in a loop reactor. Especially a suitable inert low-boiling hydrocarbon can be used as a reaction medium. Examples of such hydrocarbons are propane, butane,

20 and pentane. Also higher hydrocarbons, such as hexane, heptane, octane, cyclohexane, or cycloheptane, can be used. As disclosed in WO-A-92/12182, special advantages can be achieved, if the liquid medium is in supercritical state, i.e. the temperature in the reactor exceeds the critical temperature of the fluid mixture and the pressure in the reactor exceeds the critical pressure of the fluid mixture. 25 Especially propane can be used as a diluent in supercritical operating conditions.

When e.g. bimodal polyethylene is produced, it is common to produce the low molecular weight component in the slurry stage. Hydrogen is used to control the molecular weight. To produce the low molecular weight component, a relatively high hydrogen concentration is needed. Thus, the hydrogen to ethylene molar ratio

30 in the slurry reactor is typically between 200-800 mol/kmol, depending on the requirements of the product to be produced. It is also possible to produce the high molecular weight component in the first stage. This is usually the case for instance

when bimodal polypropylene is produced. In this case hydrogen is not necessarily present in the first stage.

The second polymerization stage in accordance with the invention is conducted in a gas phase. Preferably the second polymerization stage is conducted in a gas 5 fluidized bed reactor. Such a reactor comprises a gas inlet at the bottom of the reactor, a gas distributor plate above the gas inlet, a bed of polymer particles above the gas distributor plate, and a gas outlet at the top of the reactor. It also comprises an inlet for polymer particles from the previous polymerization stage and an outlet for removing the polymer product. Moreover, it may comprise an agitator to assist 10 in keeping the bed in a thoroughly mixed state.

The gas is introduced into the bottom of the reactor. The gas distributor plate distributes the upwardly flowing gas uniformly into the bed. The velocity of the gas needs to be such that it supports the particles forming the bed and maintains the bed in fluidized state. The unreacted gas is collected from the top of the reactor, 15 compressed and cooled. The compressed and cooled gas is reintroduced into the bottom of the reactor.

To have a more efficient cooling, the recycle gas may be cooled to such an extent that part of it is condensed, as disclosed in EP-B-89691 or EP-B-699213. The 20 condensed liquid may then be introduced into the bed as a two-phase stream containing gas and liquid, as disclosed in EP-B-89691, or the liquid may be separated from the gas stream and introduced as a separate liquid stream into the reactor, as disclosed in EP-B-699213.

When e.g. bimodal polyethylene is produced, the high molecular weight component 25 is usually produced in the gas phase reactor. Then, relatively low concentrations of hydrogen are needed. Depending on the requirements of the final product, the required hydrogen to ethylene molar ratio may be as low as 5 mol/kmol. When products having a lower viscosity are produced, the hydrogen to ethylene ratio may be about 100 mol/kmol.

In the first stage reactor, the polymer produced in that reactor and the first liquid 30 medium, comprising the diluent with monomer(s) and hydrogen dissolved therein, form a slurry. Said polymer slurry is then preferably led into a suitable separation apparatus, preferably on the top of a washing apparatus. A suitable second liquid medium is introduced into the bottom of the apparatus. The solid polymer moves downwards to the bottom, and the liquid moves countercurrently upwards. A stream

consisting mainly of liquid together with reactants and polymer dissolved therein is collected from the top of the apparatus and a stream consisting mainly of concentrated polymer slurry is collected from the bottom of the apparatus.

In order to achieve the desired separation i.e., to prevent hydrogen and other reactants to be carried over from the slurry reactor into the gas phase reactor, the volume of the second liquid medium must be at least 0.5 times the volume of the slurry from the first polymerization stage, and preferably at least the volume of the slurry from the first polymerization stage. This means that the volume of the second liquid medium introduced into the bottom of the washing apparatus must be at least 5 as high as the volume of the liquid taken out with the polymer from the bottom of the apparatus. After the separation, the concentrated polymer slurry is mixed with a suitable third liquid medium, and the mixture is transferred into the gas phase reactor. The first liquid medium of the first polymerization stage together with a 10 part of the second liquid medium is removed from the top of the apparatus. The 15 liquid collected from the top of the washing apparatus is preferably recycled into the first polymerization stage.

Any suitable washing or extraction apparatus can be used, e.g. such a plain wash column as has been suggested in US-A-4121029 to be used in a pipeloop reactor system for recovery of soluble catalysts components from polymer slurry effluent. 20 This column has a settling leg, into which the particles settle because of their higher density. The second liquid medium is preferably fed into the bottom of the column at a rate which is essentially lower than the settling rate of the particles. A column provided with a mixing apparatus can also be used, especially if it can be expected that the polymer will attach to the walls of the column.

25 The process according to the invention has also a benefit of avoiding the carry-over of the reactants from the first polymerization stage into the second polymerization stage.

The liquid medium forming the slurry in which the polymer is introduced into the gas phase reactor preferably evaporates in that reactor, thus cooling the reactor. 30 Such cooling is effective and leads to a well controlled temperature throughout the reactor.

As disclosed above, the first liquid medium comprises the diluent used in the slurry polymerization, together with monomer, eventual comonomers and optionally hydrogen dissolved therein. The second liquid medium introduced into the bottom

of the washing apparatus is preferably pure diluent used in the slurry polymerization stage, possibly containing traces of monomer(s) and hydrogen. The third liquid medium which is used to assist the transfer of the slurry into the gas phase reactor is preferably a condensate which has been obtained by condensing a part of the 5 recycle gas flow of the gas phase reactor.

The temperature and pressure in the washing apparatus are selected so that the fluid within the apparatus remains as liquid. The temperature is preferably lower than the operating temperature of the gas phase reactor, that is usually lower than 80 °C. The lower limit may be chosen based on economical considerations. A suitable range of 10 temperature of the washing apparatus is between 40 °C and 80 °C.

The operating pressure of the washing apparatus should be lower than the operating pressure of the slurry reactor but higher than the operating pressure of the gas phase reactor. Moreover, the operating pressure should be selected so that no significant flashing occurs in the slurry reactor discharge line. A suitable range of operating 15 pressure is from about 5 to 25 bar lower than the operating pressure in the slurry polymerization stage, or from 30 to 55 bar.

It is also possible, within the scope of the invention, to feed the second liquid medium in the middle zone of the washing apparatus and the third liquid medium into the bottom of the washing apparatus. This method of operation has the 20 advantage of dividing the washing apparatus into two zones: an upper zone with an upward liquid flow and a lower zone with a downward liquid flow. The major part of the first liquid medium introduced together with the polymer into the top of the apparatus is then discharged from the top of the washing apparatus. The concentrated polymer slurry in the third liquid medium is discharged from the 25 bottom of the washing apparatus. The carry-over of hydrogen and other reactants from the loop reactor to the bottom of the washing apparatus, and on the other hand, the carry-over of comonomer and other reactants from the gas phase reactor to the top of the washing apparatus is greatly reduced by employing this mode of operation.

30 Some undesired components, such as waxes, are also formed in the first stage. Such soluble components do not accumulate in the washing apparatus but are removed together with the first stage liquid medium. When the liquid medium is recycled into the first polymerization stage, the waxy components are preferably removed by evaporation.

Less reacted fine particles from the first stage are also removed with the first stage liquid medium from the separation apparatus. This is also advantageous since such fine particles may cause inhomogeneity in the powder of the gas phase reactor. Such particles can also be removed so as to not enrich in the slurry reactor.

- 5 Also, eventual soluble catalyst components, e.g. aluminium alkyls used as cocatalysts, are removed with the first stage liquid medium from the separation apparatus. Thus such catalyst components can be replaced by another ones in the second stage.
- 10 The polymerization stages are preferably carried out according to the description in WO-A-92/12182 (corresponds to FI-A-906428), WO-A-96/18662 (corresponds to FI-A-945926), WO-A-96/18662 (corresponds to FI-B-96216), or EP-A-688794 (corresponds to FI-A-942949).

The enclosed drawing describes two-stage processes for preparing polyethylene.

Figure 1 describes one preferred embodiment of the invention.

- 15 Ethylene, hydrocarbon medium (propane), hydrogen, and optionally comonomer are fed through a line 1 into a loop reactor 2. Solid catalyst (and possible cocatalyst) is fed through a separate line 3. In the loop reactor the reaction mixture is circulated by suitable means. The reactor is suitably cooled. The reactor can be also otherwise controlled. E.g., the reaction mixture can be analyzed, and the amount of the
- 20 possible cocatalyst can be adjusted.

From the bottom of the loop reactor 2, the reaction mixture is fed through a line 4 on the top of an extraction column 5 provided with a mixer. In the column, the solid polymer containing the catalyst moves downwards to the bottom. Dispersion medium is fed through a line 6 on the bottom of the column, and the polymer is further transferred with the dispersing medium through a line 7 into a fluidized bed reactor 8. The dispersion medium evaporates in the reactor. Ethylene, and possible other components, such as comonomer, nitrogen, and hydrogen, are fed into the reactor through a line 9. The polymer particles form a bed on the gas distribution plate of the reactor. The bed is maintained in a fluidized state by introducing an upwardly flowing gas flow from the bottom of the reactor through the gas distribution plate. Unreacted gas is taken from the top of the reactor through a line 10. The gas is cooled in a cooler 11 so that part of it condenses. The partially condensed gas flow is further led into a tank 12. The noncondensed part of the gas flow is fed from the tank 12 through a line 13 to the bottom of the reactor.

The condensed part of the gas flow is led from the tank 12 via a conduit 6 to the bottom of the extraction column 5 to be used as the dispersion medium.

Polymer is removed from the gas phase reactor 8 through a line 14.

5 The hydrocarbon medium is removed from the top of the extraction column 5 through a line 15. Waxes are removed as dissolved in the hydrocarbon. Also finest particles are removed together with the hydrocarbon flow. The hydrocarbon may be separated from the waxes in an evaporator 16, and most of the hydrocarbon is recycled through a line 17 into the loop reactor 3. Part of the hydrocarbon is fed through a hydrogen separator 18 and line 19 as a wash liquid above the dispersion 10 medium into the extraction column. It is preferable that the mixing of the wash liquid and the dispersion medium is avoided as far as possible.

15 At the bottom of the extraction column 5, some reaction medium of the loop reactor may be mixed with the condensate led to the bottom. However, the amount can be kept so slow that it has in practice no effect in the gas phase reactor. Correspondingly, some condensate may be mixed with the liquid removed from the top of the column, but also this amount can be kept so slow as to not have any effect.

According to one embodiment of the invention, the top of the column 5 may have the shape of a hydrocyclone to enhance the separation.

20 According to one embodiment, a cocatalyst is used in the loop reactor, and this cocatalyst is separated from the polymer together with the reaction medium. Different cocatalyst can then be used in the gas phase reactor. In a typical arrangement, about 1/30 - 1/5 of the cocatalyst used in the slurry reactor can be removed.

25 According to another preferred embodiment of the invention, in the process of Fig. 1, the second liquid medium is introduced into the middle zone of the column 25.

30 The introduction of the liquid into the middle zone divides the column into two zones. In the upper zone there is an upward liquid flow and in the lower zone there is a downward liquid flow. The upwardly flowing liquid, which contains the major fraction of the first liquid medium comprising the diluent, ethylene and hydrogen from the loop reactor, is collected from the top of the column via conduit 15. The downwardly flowing liquid, which contains the major fraction of the third liquid

medium comprising the condensed part of the recycle gas stream, is collected from the bottom of the column via the conduit 7.

### Examples

The following Examples describe the background of the invention and its benefits.

5 Examples 4 to 6 show the influence of flashing especially on the particle size distribution.

MFR was measured according to ISO 1133, at 190 °C.

Particle size distribution was determined by laser diffraction method, using an equipment by Malvern. The fines were considered as particles having a diameter 10 smaller than 100 microns.

#### Example 1

15 Into a loop reactor having volume of 57 m<sup>3</sup> catalyst, triethyl aluminum (as cocatalyst), ethylene, propane and hydrogen are introduced so that the production of polymer is 7 tons/h and solids concentration at the reactor outlet is 45%. Thus, the mass flow of slurry out of the reactor is 16 tons/h. The amount of dissolved polymer in the fluid phase is 1.6%, corresponding to a concentration of 8.3 kg/m<sup>3</sup>. The concentration of triethyl aluminum in the fluid is 30 g/m<sup>3</sup> (corresponding to 24 ppm Al in polymer).

20 The slurry is then introduced into the top of an extraction column operating at 45 °C, where it is flushed with a fresh diluent fed from the bottom of the column. The volumetric flow rate of slurry at this temperature is 24 m<sup>3</sup>/h. The density of the fluid phase is 520 kg/m<sup>3</sup>.

25 The volumetric flow rate of the fresh wash diluent is 0.5 times the volumetric flow rate of slurry, i.e. 12 m<sup>3</sup>/h. The diameter of the extraction column is 1.5 m and the length is 6 m.

30 In these conditions, the dissolved polymer and the triethyl aluminum are removed from the polymer flow to such an extent that the concentration of dissolved polymer is 1.7 kg/m<sup>3</sup> at the bottom of the column and the concentration of triethyl aluminum is 6.1 g/m<sup>3</sup> (corresponding to 5 ppm Al in polymer). The data is summarized in Table 1.

**Example 2**

The process according to the Example 1 is operated in otherwise similar manner, except that the ratio  $r$  of volumetric feed flow of wash diluent to the volumetric flow of polymer slurry is 1.0 instead of 0.5. The results are shown in Table 1.

5 **Example 3**

The process according to the Example 1 is operated in otherwise similar manner, except that the ratio  $r$  of volumetric feed flow of wash diluent to the volumetric flow of polymer slurry is 0.75 instead of 0.5. The results are shown in Table 1.

**Table 1**

Example	$r$	Dissolved polymer		TEA			
		Inlet	Outlet	Inlet		Outlet	
		kg/m <sup>3</sup>	kg/m <sup>3</sup>	g/m <sup>3</sup>	ppm	g/m <sup>3</sup>	ppm
1	0.5	8.3	1.7	30	24	6	5
2	1.0	8.3	0.3	30	24	1	1
3	0.75	8.3	0.7	30	24	3	2

10

**Example 4 (comparative)**

Ethylene was continuously polymerized at a rate of 25 kg/h in a 500 dm<sup>3</sup> loop reactor operating at 95 °C temperature and 60 bar pressure in the presence of a catalyst prepared according to Example 3 of EP-688794. The slurry containing the

15 liquid medium and the polymer were discharged intermittently from the reactor to a flash tank operating at 3 bar pressure. The composition of the liquid medium was analyzed and it was found to contain about 88% by mole propane, 7% by mole ethylene and 3% by mole hydrogen. The remaining 2% contained aliphatic hydrocarbons, like methane, ethane, n-butane and isobutane.

20 A polymer sample was taken from the flash tank. The particle size distribution was measured. It was found that the fraction of particles having a diameter less than 100 microns was about 15%.

The polymer was transferred from the flash tank into a gas phase reactor. The polymerization was continued there.

**Example 5**

Ethylene was continuously polymerized as in Example 4. The slurry containing the liquid medium and the polymer were discharged intermittently from the reactor without a flashing stage directly to the gas phase reactor. The composition of the liquid medium was analyzed and it was found to contain about 88% by mole propane, 7% by mole ethylene and 3% by mole hydrogen. The remaining 2% contained aliphatic hydrocarbons, like methane, ethane, n-butane and isobutane.

A polymer sample was taken from the slurry into a 3 dm<sup>3</sup> stainless steel sample container having valves at both ends. The liquid medium was slowly removed from

the sample by evaporating the medium during a period of 30 minutes by carefully opening one of the valves. The particle size distribution of the polymer was measured. It was found that the fraction of particles having a diameter less than 100 microns was about 6%. Thus, this example shows that the level of fines can be significantly reduced if the flashing stage can be avoided.

**15 Example 6 (comparative)**

To further illustrate the effect of flashing and especially the pressure in the flash stage, an experiment was made where different flashing pressures were tested. This illustrates the effect of flashing at a higher pressure, or a case where flashing does not take place until in the gas phase reactor.

**20 The polymerization in the loop reactor was done according to Example 4.**

Run	A	B	C	D
Flash pressure, bar	3	10	15	20
MRF <sub>2</sub> of loop polymer, g/10 min	220	320	230	230
Fines, %	19	14	12	13
APS, mm	0.22	0.25	0.26	0.26

**Claims**

1. A method for transferring polymer from a slurry reactor into a gas phase reactor, in said slurry reactor the polymer being suspended in a first liquid medium, characterized in that said polymer suspended in said first medium is separated from said first medium, washed with a second liquid medium, mixed with a third liquid medium, and transferred with said third liquid medium into said gas phase reactor.  
5
2. A polymerization process, in which monomer is first polymerized in a slurry reactor where formed polymer is suspended in a first liquid medium, and thereafter led into a gas phase reactor where the polymerization is continued, characterized in that said polymer from said slurry reactor is separated from said first liquid medium and suspended in a second liquid medium, separated from said second liquid medium, suspended in a third liquid medium, and transferred with said third liquid medium into said gas phase reactor.  
10
3. A process according to claim 2, wherein said third liquid medium is evaporated in said gas phase reactor.  
15
4. A process according to claim 3, wherein gas from said gas phase reactor is removed and condensed, and the condensate is used as said third liquid medium.
5. A process according to any of claims 1-4, wherein said polymer from the slurry reactor is separated from said first liquid medium in a washing apparatus.
- 20 6. A process according to any of claims 1-5, wherein, after said first liquid medium has been separated, waxes or fine particles are separated from said first medium.
7. A process according to any of claims 5-6, wherein said separated first liquid medium is used as a wash liquid in said washing apparatus.
- 25 8. A process according to claim 7, wherein hydrogen said first liquid medium contains hydrogen, and said hydrogen is removed from said first liquid medium before using it as a wash liquid.
9. A process according to any of claims 1-8, wherein said slurry reactor is a loop reactor.
- 30 10. A process according to any of claims 1-9 for polymerizing or copolymerizing olefins.

11. A polymerization plant, comprising
  - a first reactor, which is a slurry reactor for polymerizing monomer in a first liquid medium,
  - a separation apparatus for separating said first liquid medium from the polymer obtained from said slurry reactor,
  - means for mixing the separated polymer with a second liquid medium,
  - means for separating said polymer from said second liquid medium,
  - means for mixing said polymer with a third liquid medium,
  - a second reactor, and
- 10 - means for transferring said polymer with said third liquid medium into said second reactor.

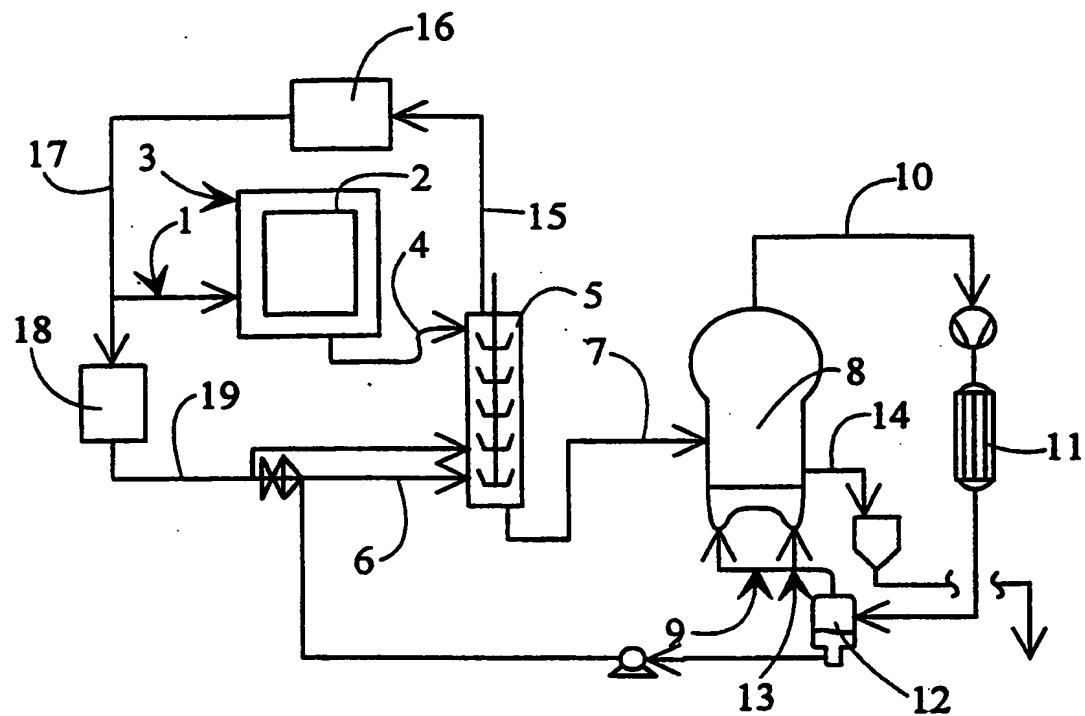


Fig. 1

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 00/00032

## A. CLASSIFICATION OF SUBJECT MATTER

IPC7: C08F 2/00

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: C08F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4692501 A (HIDEO MINESHIMA ET AL), 8 Sept 1987 (08.09.87), column 10, line 10 - line 28, figure; claims ---	1-11
A	WO 9859002 A1 (BOREALIS A/S), 30 December 1998 (30.12.98), page 3, line 17 - line 30 ---	1-11
A	US 5175208 A (TADASHI ASANUMA ET AL), 29 December 1992 (29.12.92), column 6, line 64 - column 7, line 27 --- -----	1-11

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Information on patent family members

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